(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: 0 248 119 A1

(12) EUROPEAN PATENT APPLICATION

(21) Application number: 86200950.3

(22) Date of filing: 02.06.86

(51) Int. Cl.[4]: G03G 13/09

(43) Date of publication of application: 09.12.87 Bulletin 87/50

(84) Designated Contracting States: AT BE CH DE FR GB IT LI LU NL SE

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(54) Improved method for the development of electrostatic images.

(57) A method for the development of a multiplicity of electrostatic images by the use of a developer material comprising a mixture of electrostatically charged toner and carrier particles, wherein in course of such use for replenishment fresh toner is added to the residual material of the batch, characterised in that the developer, being called start developer, used for the development in the first copying cycle of a running-in period of development is a batch of developer comprising a toner-carrier mixture wherein the toner particles, called start toner particles have a mean particle diameter smaller than the mean particle diameter of toner particles added in said replenishment, and wherein the difference in mean particle diameter of said toner particles is such that the apparent density (A.D.) of the start developer and of the toner-carrier mixture of use upon said replenishment is differing by not more than 10 %, the composition of the toner particles used in the start developer being the same as the composition ofthe toner particles used in the replenishment.

IMPROVED METHOD FOR THE DEVELOPMENT OF ELECTROSTATIC IMAGES

The present invention relates to an improved method for the development of electrostatic images by means of a developer material comprising a mixture of electrostatically charged toner and carrier particles.

As is well known, electrostatic images can be formed in various ways, e.g. by electrography, ionography or electrophotography. In this specification particular embodiments of the invention will be described for the development of electrophotographically formed images but the invention can equally well be applied for developing electrostatic images formed in some other way.

When using a dry developer comprising a mixture of toner and carrier particles, frictional contact between the different types of particles causes them to acquire electrostatic charges of opposite sign due to the triboelectric effect. The polarity of the toner particles is appropriate for causing their image-wise deposition on the electrostatic image to be developed. This electrostatic image can for example be formed on a photoconductive layer simply by overall charging the layer and then image-wise exposing it to activating electromagnetic radiation, e.g. light or X-rays through a graphic original. In that case residual charges remain which are not dissipated by the irradiation and form the electrostatic image. In the case that the photoconductive layer was exposed through a positive graphic original the development step will produce a positive copy of the original. Alternatively charges opposite in polarity to the said residual charges can be induced in the exposed areas of the photoconductive layer and development can occur by deposition of toner particles responsive to the induced charges. This procedure, known as reversal development, is useful for producing positive copies of negative graphic originals through which the photoconductive layer is exposed, or positive copies of images traced on the photoconductive layer by an image-wise modulated scanning laser beam or by points of light emitted from an exposure head comprising an array of selectively image-wise addressed light-emitting diodes.

For developing an electrostatic image by means of a dry developer material of the kind hereinbefore referred to, there are various known ways of bringing the developer material into contact with or into the immediate vicinity of the electrostatic image. Cascade development and magnetic brush development are two examples. These and other development techniques are described in detail by Thomas L. Thourson in "Xerographic Development Processes : A Review" - IEEE Transactions on Electron Devices, Vol. ED-19, No. 4, April 1972, pp. 497-504.

The present invention is particularly but not exclusively suitable for use in the development of electrostatic images by means of a magnetic brush technique. In magnetic brush development, use is made of a developer material comprising magnetically susceptible carrier particles, to which smaller toner particles electrostatically adhere. The developer material is brought to the electrostatic image by a magnetic applicator which may for example comprise a rotatable non-magnetic cylinder housing one or more magnets. The magnetic carrier particles are attracted to the applicator by the surrounding magnetic field and are thereby held around the applicator in a loose, brush-like orientation during the development cycle. When this brush-like mass of carrier particles with adhering toner particles is drawn across a surface bearing a latent electrostatic image, the toner particles are electrostatically attracted to oppositely charged latent image areas to form the developed image. A bias voltage is usually applied to the magnetic applicator for restraining deposition of toner on the background areas surrounding the developed image and thus promoting clean development. When using a magnetic brush development technique, reversal development can be carried out by applying to the magnetic toner applicator an appropriate bias voltage causing it to act as a development electrode which induces toner-attracting charges in the surface on which the developer image is to be formed (cf. R.M.Schaffert "Electrophotography" The Focal Press - London, New York, enlarged and revised edition, 1975 and T.P.Maclean "Electronic Imaging" Academic Press - London 1979, p. 231).

In cyclical copying processes wherein electrostatic images are successively formed and developed on an image-wise chargeable element, e.g. a photoconductive layer, from which the toner is image-wise transferred to sheet receptor material, it is common practice to derive the toner for successive developments from a common batch of the developer material held in a reservoir. During use of a given batch of developer material it undergoes changes which affect its development capability. During an initial period of use (hereafter called the "running-in period") toner becomes smeared over the surfaces of the carrier particles and these become progressively coated with toner up to a certain thickness. This phenomenon results in changes in the triboelectric effect, the flow properties of the developer material and the

charge/mass ratio of the toner particles. Additionally of course the toner particle content of the developer material batch progressively decreases in consequence of the consumption of toner for the development of the electrostatic images. The foregoing changes in the developer material batch inevitably result in changes in the quality of the developed images unless corrective measures are taken.

Toner depletion in the batch of developer material can be compensated for by the addition of fresh amounts of toner. European Patent Application 0 140 996 describes a process wherein the toner replenishment is controlled automatically in function of electronic signals generated in dependence on deviations of the magnetic density of the toner-carrier mixture from a predetermined reference value. This method involves a lot of electronic measurements and control functions and in practice requires the use of a microprocessor. Moreover the magnetic density of the developer material is not always a reliable indicator of the developing capability of the material. This appears particularly to be the case over the course of the running-in period of the developer.

The published European Patent Application 0 154 041 describes a method for compensating for changes in the development capability of a given batch of developer material comprising magnetically susceptible carrier particles, in course of its use for developing electrostatic images on a photoconductive layer. The method makes use of a magnetic brush applicator whose bias voltage with respect to an electrically conductive backing of the photoconductive layer is controlled to effect the required compensation. The bias voltage is controlled by signals from electronic control means. This control means is fed with signals representing the number of copying cycles in which the developer batch is used and is programmed on the basis of experimental data which records changes in the development capability of the developer in function of the number of copying cycles. These data are obtained by subjecting a test batch of the developer to use conditions simulating those of the actual developer during and subsequent to its running-in period. This prior art method likewise involves a lot of electronic measurements and control functions and in practice requires the use of a microprocessor and an appreciable amount of programming software.

The present invention provides a method whereby changes in the development capability of a batch of developer material starting with a fresh mixture of toner and carrier particles are compensated for by using at the start toner having a particular size characteristic. The method compensates for changes in the development capability of the developer during its running-in period.

The invention is based upon appreciation of the fact that the progressive coating of carrier particles with toner during the running-in period results in alteration of the apparent density (A.D) of the developer batch, and on the discovery that by addition of toner of a size such that the added toner counters or reduces this alteration, the quality of the developed images produced by a multiplicity of copying cycles can be kept more uniform.

According to the present invention there is provided a method for the development of a multiplicity of electrostatic images by the use of a developer material comprising a mixture of electrostatically charged toner and carrier particles, wherein in course of such use for replenishment fresh toner is added to the residual material of the batch, characterised in that the developer, being called start developer, used for the development in the first copying cycle of a running-in period of development is a batch of developer comprising a toner-carrier mixture wherein the toner particles, called start toner particles, have a mean particle diameter smaller than the mean particle diameter of toner particles added in said replenishment, and wherein the difference in mean particle diameter of said toner particles is such that the apparent density (A.D.) of the start developer and of the toner-carrier mixture of use upon said replenishment is differing by not more than 10 %, the composition of the toner particles used in the start developer being the same as the composition of the toner particles used in the replenishment.

The invention enables the obtaining of a reproducible image quality already from the start with a new batch of developer without very sophisticated and expensive control apparatus.

For a given development process in a given machine an appropriate particle diameter of replenishment toner can be determined empirically. The replenishment toner can be added as required to keep the developed image density within acceptable limits. The dosing of replenishment toner can be effected on the basis of image density assessment made visually or by automatic sensing means as described e.g. in published German Patent Application (DE-OS) 3,301,142.

The method according to the invention is particularly beneficial for developing screened (half-tone) electrostatic images to form in a reproducible way high quality half-tone prints.

According to an embodiment the method of the present invention is modified in that the first copy is made with toner particles of said start developer in admixture with coarser toner particles used later on in the toner replenishment, with the proviso that the A.D. deviation of the resulting toner/carrier mixture calculated as the difference of the A.D. values respectively at the beginning and at the end of a running-in period corresponding with 4,000 copying cycles does not surpass 10 %.

The mean particle diameter is derived from the mean volume size which is determined by the Coulter Principle which is the subject of British Standard 3406 : Part 5 : 1983 and described in US-P 2,656,508. The Coulter Principle measurement operates with an electric current path of small dimensions which is modulated by momentary passage of each particle one-by-one. Particles suspended in an electrolyte are forced through a small aperture across which an electric current path has been established. Each particle displaces electrolyte in the aperture producing a pulse equal to its displaced volume. Thus, three dimensions or particle volume response is the basis of the Coulter Principle measurement. In the determination of the mean volume of the toner particles the COULTER COUNTER (registered trade name of Coulter Electronics Ltd, Northwell Drive, Luton, England) Model TAII/PCA 1 has been used.

The mean particle diameter ($\bar{d}$) is calculated from the mean volume size ($\bar{V}$) by the following equation :

$$\bar{d} = (6.\bar{V} /pi)^{-3}$$

wherein pi is 3.1415... .

The "apparent density" of a batch of developer material is equal to the mass (expressed in grams) per unit volume (expressed in cm3) and is measured in the following manner :

500 g of a developer composition comprising carrier and toner particles are put in a brass funnel being electrically grounded and having the following dimensions: a cylindrical upper part having a height of 73 mm and inner diameter of 50 mm. The cylindrical part ends in a conical part (angle of conicity 52°) with a height of 47 mm having at the tapering end a cylindrical closable outlet channel having a diameter of 4 mm and lenght of 20 mm. The developer composition is allowed to flow by gravity from said funnel into a graduated cylindrical measuring glass having a height of 36 cm and diameter of 4 cm. The graduation marks on the cylinder indicate increments in volume of 2 cm3.

The apparent density (A.D.) is calculated by the following equation :

$$A.D. = \frac{500}{\frac{Vmin + Vmax}{2}} \quad g/cm^3$$

wherein : Vmin is the minimum volume in cm3 of the developer corresponding with the minimum height in the cylinder,
Vmax is the maximum volume in cm3 of the developer corresponding with the maximum height in the cylinder.

Minimum and maximum volume are measured because in streaming down into the cylinder the developer composition is not accumulating in water-level fashion.

The influence which a change in the apparent density of a developer batch has on its development capability is particularly marked in the case that the developer material comprises magnetically susceptible carrier particles and is brought to the electrostatic image by a magnetic brush applicator. That is why the invention is of particular benefit when applied in that type of development technique. In certain embodiments of the invention, of that kind, use is made of a developer comprising iron bead carrier particles having a specific gravity of about 7.7 and toner particles having a specific gravity of about 1.2.

The addition of a quantity of replenishment toner can be effected one or more times during the development of a multiplicity of latent images by the developer batch. The number of replenishment doses to be added depends on the quality control standard to be observed.

The apparent density of the developer batch (toner plus carrier particles) changes over the running-in period as already referred to. At the end of the running-in period the apparent density of the developer using the same toner batch in replenishment levels off and it enters a steady-state phase. This phase normally corresponds with the production of at least 50,000 developed images. Depending on the characteristics of a given copying process and the copying machine used, the running-in period may correspond with the development of a few thousand, say 4000, electrostatic images. Therefore it will often not be considered satisfactory to delay addition of toner particles with larger diameter until the end of the running-in period. Preferably a dose of such replenishment toner particles is added one or more times during the running-in period.

The replenishment with toner at any given replenishment time is preferably such that the toner-concentrations of the batch before and after replenishment do not differ by more than 10 %.

Preferably, the toner/carrier weight ratio in the developer batch at the commencement of its use is in the range 0.044 to 0.048 and the ratio is maintained in that range by toner replenishment both during the running-in period and subsequently.

The mean particle diameter of the replenishment toner is preferably not in excess of 30 um. The mean particle diameter of the start toner particles is preferably at least 2 um smaller than, and more preferably at least 5 um smaller than that of the replenishment toner.

The increase in the apparent density of the developer batch which occurs during the running-in period tends to be greater the larger are the toner particles. Large toner particles before the smearing takes place on the carrier particles are piled up with relatively large air voids inbetween. From this standpoint it is advantageous for the start toner particles in the batch at the commencement of its use to be relatively small. Preferably they have a mean particle diameter in the range from 3um to 8 um. And the particles of the replenishment toner preferably have a mean particle diameter in the range from 10 to 12 um. Such toner particles fractions can be obtained by size classification of a common supply of particulate toner, obtained e.g. by grinding of a solidified melt. The particle size classification can be carried out by means of an air sifter or hydrocyclone.

For a given charge density of the charge-carrying surface the maximum development density attainable with toner particles of a given size is determined by the charge/toner particle mass ratio, which is determined substantially by the triboelectric charge obtained by friction-contact with the carrier particles.

On using a carrier-toner mixture the development of a latent electrostatic charge pattern is carried out e.g. by cascade-, or magnetic brush development which techniques are described in detail by Thomas L. Thourson in his article "Xerographic Development Processes : A Review", IEEE Transactions on Electron Devices, Vol. ED-19, No. 4, April 1972, p. 497-504.

A preferred toner composition for use according to the present invention in magnetic brush development is described in US-P 4,525,455.

Suitable carrier particles for use in cascade development are e.g. glass beads and for magnetic brush development are magnetizable beads, e.g. iron beads as described e.g. in the United Kingdom Patent Specification 1,438,110.

The carrier particles are preferably at least 3 times larger in size than the toner particles and more preferably have an average grain size in the range of 50 to 1000 microns. Preferably magnetic or magnetizable carrier particles, e.g. iron or steel beads of 300 to 600 microns are used. The developer composition may for example contain 0.1 to 10 parts by weight of toner particles per 100 parts by weight of carrier particles. The iron or steel beads may be subjected to special pretreatments to enhance the triboelectric charging of the toner. Suitable coating-treatments of carrier beads are described e.g. in said last mentioned U.K. Patent Specification.

Use can be made of iron carrier particles that have been washed free from grease and other impurities and which have a diameter of 1.52×10^{-1} to 2.03×10^{-1} mm as referred to in United States Patent 2,786,440.

Preferred carrier beads have almost a spherical shape and are prepared e. g. by a process as described in United Kingdom Patent Specification 1,174,571.

In order to improve the flowing properties of the developer the toner particles are mixed with a flow improving means such as colloidal silica particles and/or microbeads of a fluorinated polymer. The flow improving means is used e.g. in an amount of 0.05 to 1 % by weight with respect to the toner.

Colloidal silica has been described for that purpose in the United Kingdom Patent Specification 1,438,110 e.g. AEROSIL 300 (trade mark of Degussa, Frankfurt (M) W.Germany for colloidal silica having a specific surface area of 300 sq.m/g). The specific surface area can be measured by a method described by Nelsen and Eggertsen in "Determination of Surface Area Adsorption Measurements by Continuous Flow Method", Analytical Chemistry, Vol. 30, No. 8 (1958) 1387-1390.

A fluorinated polymer useful for flow improvement of the toner and carrier particles is polyvinylidene fluoride used in the form of beads having an average particle size of 5 um sold under the trade name KYNAR RESIN 301 by Pennwalt Corp. - Plastic div. England.

Other suitable fluorinated polymer beads for improving the flowing properties of the toner as well as of the carrier particles are described in the United States Patent Specification 4,187,329. A preferred fluorinated polymer for said use is poly(tetrafluoroethylene) having a particle size of 3 to 4 um and melting point of 325-329°C. Such poly(tetrafluoroethylene) is sold under the trade name HOSTAFLON TF-VP-9202 by Farbwerke Hoechst A.G. W.Germany.

The colloidal silica and at least one of said fluorinated polymers are preferably mixed with the toner in a proportion of 0.15 % and 0.075 % by weight respectively. The toner becomes thereby non-tacky and obtains a reduced tendency to form a film on the xerographic plates or drums e.g. having a vapour-deposited coating of a photoconductive Se-As alloy on a conductive substrate e.g. aluminium.

In order to obtain toner particles having magnetic properties a magnetic or magnetizable material may be added during the toner production.

Magnetic materials suitable for said use are magnetic or magnetizable metals including iron, cobalt, nickel and various magnetizable oxides including Fe_2O_3, Fe_3O_4, CrO_2, certain ferrites derived from zinc, cadmium, barium and manganese. Likewise may be used various magnetic alloys, e.g. permalloys and alloys of cobalt-phosphors, cobalt-nickel and the like or mixtures of any of these. Good results can be obtained with about 10 % to about 80 % by weight of magnetic material with respect to the resin binder.

The following examples 1 and 2 wherein example 1 is based on comparative tests showing apparent density changes illustrate the present invention. All parts and percentages are by weight unless otherwise indicated.

EXAMPLE 1

Iron carrier beads of a diameter of 65 um having a thin iron oxide skin are mixed with 4.6 % of toner particles prepared according to the method described in US-P 4,525,445 proceeding as follows:

90 parts of ATLAC T500 (trade name for a propoxylated bisphenol A fumarate polyester of Atlas Chemical Industries Inc., Wilmington - Delaware, U.S.A.), 5 parts of carbon black (Spezialschwarz IV - trade name) and 5 parts of a nigrosine base neutralized with stearic acid were mixed in a heated kneader. The melting range of ATLAC T500 (trade name) was 65-85°C. The melting range of the nigrosine base stearic acid salt was 110-120°C. The mixing proceeded for 15 minutes at a temperature of the melt corresponding with 105°C. Thereafter the kneading was stopped and the mixture was allowed to cool to room temperature (20°C). At that temperature the mixture was crushed and milled to form a powder. From the obtained powder, the particles with a size between 3 and 30 um were separated to form the toner.

By means of an air-sifter two batches of toner particles were separated having respectively a mean particle diameter of 11 um and of 5 um respectively. The particle size measurement was carried out with a COULTER COUNTER (trade name) Model TAII/PCA 1 particle size analyzer operating on the above explained Coulter Principle.

For carrying out different tests these toner particles (T) were mixed with iron beads (C) coated with a thin iron oxide skin and having a particle size of 65 um at a ratio indicated in the Table hereinafter.

The mixing was carried out in a cylindrical container having an inner diameter of 178 mm and a lenght of 200 mm. The mixing proceeded by rotating said container at 57 cycles/minute during a period of 5 minutes. The apparent density (A.D.) of the obtained carrier/toner mixtures is listed in said Table under the wording START. In the same Table the apparent density (A.D.) obtained after 4,000 copies in a copying machine is given under the wording AFTER.

TABLE

Test No.	T/C	% toner 11 um	% toner 5 um	Start A.D. g/cm3	After A.D. g/cm3
1	4.6/100	100	-	2.85	3.30
2	id.	-	100	3.11	3.29
3	id.	50	50	3.11	3.30
4	id.	75	25	2.87	3.31
5	id.	95	5	2.86	3.30

EXAMPLE 2

As start developer the toner/carrier mixture of test no. 2 of Example 1 was used in a GEVAFAX X-12 (trade name) electrophotographic copying apparatus operating with magnetic brush development in the reversal mode.

Replenishment was started already after the first 20 copies with toner particles of test no. 1 of Example 1 and further copying with regular replenishment to keep constant developer level in the developer tray (level detection as described in DE-OS 3,301,142) was carried out for about 50,000 copying cycles. About 100 mg of toner particles was used per copy and at the start of developing 30 g of toner were present in the batch of the start developer.

From visual inspection of the copies obtained in the different copying cycles from the start on, using in the exposure a graphic art DIN A4 10 % black original, it was concluded that the image quality remained constant.

For the next 50,000 copying cycles there is started again with a fresh toner/carrier mixture of Test 2 of Example 1 and replenishment is carried out with a fresh toner carrier mixture of Test 1 of Example 1.

Claims

1. A method for the development of a multiplicity of electrostatic images by the use of a developer material comprising a mixture of electrostatically charged toner and carrier particles, wherein in course of such use for replenishment fresh toner is added to the residual material of the batch, characterised in that the developer, being called start developer, used for the development in the first copying cycle of a running-in period of development is a batch of developer comprising a toner-carrier mixture wherein the toner particles, called start toner particles, have a mean particle diameter smaller than the mean particle diameter of toner particles added in said replenishment, and wherein the difference in mean particle diameter of said toner particles is such that the apparent density (A.D.) of the start developer and of the toner-carrier mixture of use upon said replenishment is differing by not more than 10 %, the composition of the toner particles used in the start developer being the same as the composition of the toner particles used in the replenishment.

2. A method according to claim 1, wherein use is made of a developer comprising iron bead carrier particles having a specific gravity of about 7.7 and toner particles having a specific gravity of about 1.2.

3. A method according to claim 1 or 2, wherein a dose of said replenishment toner particles is added one or more times during the running-in period of the batch of developer material.

4. A method according to any preceding claim, wherein the toner/carrier weight ratio in the developer batch at the commencement of its use is in the range from 0.044 to 0.048 and the ratio is maintained in that range by toner replenishment both during the running-in period and subsequently.

5. A method according to any preceding claim, wherein the mean particle diameter of the added replenishment toner is not in excess of 30 um.

6. A method according to claim 5, wherein the mean particle diameter of the start toner is at least 2 um smaller than that of the replenishment toner.

7. A method according to any preceding claim, wherein the start toner particles in the batch at the commencement of its use have a mean particle diameter in the range from 3 um to 8 um.

8. A method according to claim 7, wherein the particles of the replenishment toner have a mean particle diameter in the range from 10 to 12 um.

9. A method according to any of the preceding claims, wherein the carrier particles are magnetic or magnetizable.

10. A method according to claim 1, with the modification that the first copy is made with toner particles of said start developer in admixture with coarser toner particles used lateron in the toner replenishment, with the proviso that the A.D. deviation of the resulting toner/carrier mixture calculated as the difference of the A.D. values respectively at the beginning and at the end of a running-in period corresponding with 4,000 copying cycles does not surpass 10 %.

11. A method according to any of the preceding claims, wherein the development is carried out with magnetic brush.

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EUROPEAN SEARCH REPORT

Application number

EP 86 20 0950

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	PATENTS ABSTRACTS OF JAPAN, vol. 9, no. 93 (P-351)[1816], 23rd April 1985; & JP-A-59 220 765 (MITA KOGYO K.K.) 12-12-1984 * Abstract *	1,4,5 7-9	G 03 G 13/09
A	US-A-4 288 518 (MIYAMOTO) * Column 2, lines 31-39; abstract *	1,9,11	
A	IBM TECHNICAL DISCLOSURE BULLETIN, vol. 17, no. 9, February 1975, page 2674, New York, US; S.R. FULLER et al.: "Controlling developer break-in effects" * Whole document *	1	
A	PATENTS ABSTRACTS OF JAPAN, vol. 10, no. 89 (P-444)[2146], 8th April 1986; & JP-A-60 225 859 (RICOH K.K.) 11-11-1985 * Abstract *	1	TECHNICAL FIELDS SEARCHED (Int. Cl.4) G 03 G 13/00 G 03 G 15/00
A	PATENTS ABSTRACTS OF JAPAN, vol. 8, no. 153 (P-287)[1590], 17th July 1984; & JP-A-59 52 262 (MINOLTA CAMERA K.K.) 26-03-1984 * Abstract *	1	

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	02-02-1987	CIGOJ P.M.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
O : non-written disclosure
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T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons
& : member of the same patent family, corresponding document

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EUROPEAN SEARCH REPORT

Application number

EP 86 20 0950

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
A	PATENTS ABSTRACTS OF JAPAN, vol. 8, no. 169 (P-292)[1606], 4th August 1984; & JP-A-59 64 864 (KONISHIROKU SHASHIN KOGYO K.K.) 12-04-1984 * Abstract * -----	1	

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	02-02-1987	CIGOJ P.M.

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X : particularly relevant if taken alone
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D : document cited in the application
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& : member of the same patent family, corresponding document